



# Facile Solvothermal Synthesis and Characterization Studies of Pure and Pb Doped Cadmium Sulfide Nanoparticles for Potential Photovoltaic Applications

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## ABSTRACT

Pure and Pb doped cadmium sulfide (CdS) nanoparticles were synthesized using a straight forwarded solvothermal technique. The powder x-ray diffraction (PXRD) studies done on the sample synthesized revealed wrtztite phase of nanoparticles with improved crystalline properties due to the integration of dopant Pb into the host CdS. The UV – Vis characterization studies also reveal enhanced optical properties of the nanoparticles, which can be efficiently tapped for various photovoltaic applications.

**Keywords:** Cadmium sulfide nanoparticles; Doped CdS; Photovoltaic; Quantization effect; Solvothermal synthesis.

## 1. INTRODUCTION

In recent years, there has been considerable interest generated in the study of compound semiconductors with dimensions in the nanometer range. The novel optical and transport properties exhibited by these materials continuously fascinate and hold the interest of researchers worldwide. Over the last 10 years, it has been realized that nanomaterials have some unique properties, which have been attributed to their size-dependent bandgap energy (Khan *et al.* 2019b; Iqbal *et al.* 2020). Nano-semiconductor materials that exhibit peculiar properties which are rarely shown by their bulk counterparts have attracted much interest from both fundamental and technological researchers (Barglik-Chory *et al.* 2003; Priya *et al.* 2012; Loudhaief *et al.* 2018).

Metal oxide and metal sulfide semiconductor nanoparticles in their pure and doped form were synthesized and examined widely due to their intervening properties, which makes them attractive to the material scientists for their countless applications (Martínez-Alonso *et al.* 2014; Malashchonak *et al.* 2015; Ertis and Boz 2017; Khan *et al.* 2019a). Among many combinations of these materials, pure and doped metal sulfides of group II – VI elements are particularly under the limelight of researchers. These semiconductor materials have greater scope in the research arena not limited to solar photovoltaic (Priya *et al.* 2012; Martínez-Alonso *et al.* 2014; Veerathangam *et al.* 2018), photocatalytic (Chauhan *et al.* 2013; Ertis and Boz 2017),

energy materials, storage devices, biomedical engineering materials, etc. Cadmium sulfide (CdS) is one such metal sulfide semiconductor with enormous potential applications as it can be easily doped with other elements thus exhibiting a wide range of optical and electrical properties. In doped CdS semiconductors, the impurity states play a crucial role in affecting the electronic energy structures and transition probabilities in contrast with undoped ones (Nag *et al.* 2008). For doped nanocrystalline semiconductors, quantum confinement effects in the energy states also produce unusual physical and optical behaviour (Maleki *et al.* 2007; Nag *et al.* 2008; Priya *et al.* 2012; Malashchonak *et al.* 2015). The doping ions act as recombination centres for the excited electron-hole pairs and results in a display of a strong and characteristic luminescence (Barglik-Chory *et al.* 2003; Chauhan *et al.* 2013; Qi *et al.* 2015; Khan *et al.* 2019b; Khan *et al.* 2019a; Iqbal *et al.* 2020).

Arya *et al.* have prepared Cu doped CdS nanoparticles via the sol-gel method and utilized it as a photodiode (Arya *et al.* 2018). Ag-doped CdS nanoparticles were synthesized by Iqbal and his team using the chemical co-precipitation method (Iqbal *et al.* 2020). They claim that these nanoparticles are suitable for solar cell-specific applications due to their reduced bandgap and increased optical transparency. Chauhan *et al.* used Fe doped CdS (Chauhan *et al.* 2013), and Ertis and his team used different metal ion (Ni, Co, Ce, Sb) doped CdS nanoparticles (Ertis and Boz 2017) for the

photodegradation of methylene blue under visible light irradiation and found it to be useful for the same. Likewise, many other research groups have investigated the CdS nanoparticles doped with Mn (Barglik-Chory *et al.* 2003; Nag *et al.* 2008; Kim *et al.* 2018), La (Qi *et al.* 2015), Bi (Loudhaief *et al.* 2018), Gd (Khan *et al.* 2019a), etc., and studied the properties of these materials for applications not limited to solar energy conversion (Martínez-Alonso *et al.* 2014; Qi *et al.* 2015; Veerathangam *et al.* 2018; Kim *et al.* 2018; Khan *et al.* 2019b), photocatalytic degradation (Chauhan *et al.* 2013), a photodiode (Arya *et al.* 2018) and optoelectronic (Priya *et al.* 2012; Manthrammel *et al.* 2019; Khan *et al.* 2019a) device applications. More recently, Khan *et al.* have theoretically explored the optical properties of the Pb doped CdS system by interpreting computational modelling (Khan *et al.* 2019b). They have suggested that Pb enhances the optoelectrical properties of CdS lattice, and hence it can be labelled as an ideal material for photo-detector, quantum dot-based solar cells, and photosensitive applications. Many research articles have reported the synthesis of pure and doped CdS nanoparticles whereas, literature on the experimental study of Pb doped CdS nanoparticles and their properties are inadequately reported. This being the case, an attempt has been made in this research article to highlight the peculiar and fascinating experimental properties of Pb doped CdS nanoparticles.

As for CdS nanoparticles and their doping is a concern, plenty of synthesis procedures with special emphasis on key parameters have been reported to enable and fine-tune physical, chemical and optical properties of semiconductors. For example, thermal decomposition (Martínez-Alonso *et al.* 2014), laser ablation, microwave irradiation (Nabiyouni *et al.* 2013; Martínez-Alonso *et al.* 2014; Manthrammel *et al.* 2019), sonochemical (Martínez-Alonso *et al.* 2014), reverse micelles process, chemical reduction, co-precipitation method (Manthrammel *et al.* 2019; Iqbal *et al.* 2020), ultrasonic irradiation, radiolysis, solvothermal (Li *et al.* 1999; Priya *et al.* 2012; Martínez-Alonso *et al.* 2014) and electrochemical etc. In most of the above papers controlling the particle size distribution and stability has been reported to be difficult. Taking the above considerations into mind, in the present work, an attempt has made to synthesis and characterize pure and two different concentrations of Pb<sup>2+</sup> doped (4 mole% and 8 mole%) CdS nanoparticles by using one of the convenient and cost-effective simple solvothermal methods to obtain high-quality uniform size nanoparticles. The structural and optical properties of

these nanoparticles have also been analyzed for their suitability in photovoltaic applications.

## 2. EXPERIMENTAL DETAILS

### 2.1 Synthesis and Characterization of Pure and Pb doped CdS nanoparticles

A facile solvothermal technique was adopted for the synthesis of pure and Pb doped CdS nanoparticles using a conventional heating technique. An equal proportion of metal-bearing precursor cadmium acetate dihydrate ((CH<sub>3</sub>COO)<sub>2</sub>Cd.2H<sub>2</sub>O) and sulphur bearing precursor thiourea (CH<sub>4</sub>N<sub>2</sub>S) was dissolved in 300 ml of ethylene glycol and stirred well to obtain a clear solution and then heated at 200 °C using a hot plate until the solvent evaporated completely. The final colloidal precipitate obtained was washed with double distilled water and acetone several times and filtered to obtain the final product. For the synthesis of Pb doped (4 and 8 mole % ) CdS nanoparticles, desired proportion of lead acetate trihydrate was added separately to the homogeneous mixed solution of reactants and then stirred for about 1 hour. Subsequently, a similar synthesis procedure was repeated to obtain Pb doped CdS nanoparticles. The final product was collected and characterized structurally and optically. Structural characterization was carried out using powder X-ray diffraction (Panalytical X'PERT PRO). The morphology of these nanoparticles was analyzed using a field emission scanning electron microscope (TESCAN -MIRA3 XMU), and a Uv-Vis Spectrometer (Shimadzu -UV 3600Plus) was used to analyze the optical properties of these nanoparticles.

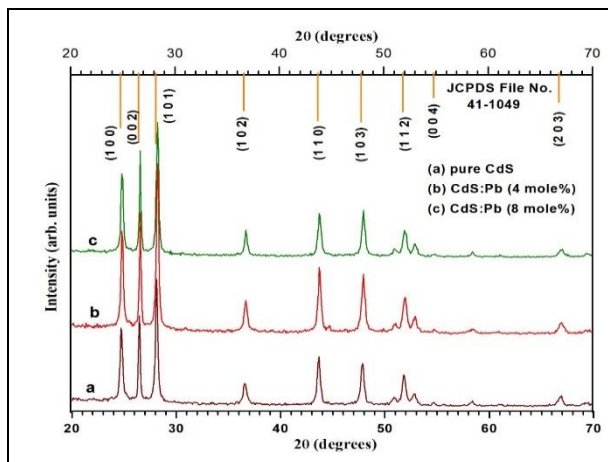
## 3. RESULTS & DISCUSSIONS

### 3.1 Structural Analysis

The obtained X-ray diffraction patterns (XRD) of pure CdS, 4 mole% Pb<sup>2+</sup> and 8 mole% Pb<sup>2+</sup> doped CdS nanoparticles are shown in Figure 1,. The diffraction peaks (1 0 0), (0 0 2), (1 0 1), (1 0 2), (1 1 0), (1 0 3), (1 1 2), (0 0 4) and (2 0 3) indicate that the synthesized pure and Pb doped CdS nanoparticles have wurtzite structure, from which the observed peaks can be indexed to the hexagonal phase of CdS with space group P6<sub>3</sub>mc. With comparison to JCPDS file No. 41-1049, the peak orientations from the spectra showed hexagonal structure of the pure and Pb doped CdS. The CdS nanoparticles' XRD pattern was compared to that seen in the literature (Maleki *et al.* 2007; Priya *et al.* 2012; Nabiyouni *et al.* 2013; Abd El-Sadek *et al.* 2019).

From the recorded spectra, one can infer that the degree of crystallinity improves with the increasing concentration of dopant in the host CdS lattices. More

interestingly, when  $\text{Pb}^{2+}$  is added into the pure CdS, no diffraction peaks corresponding to PbS/CdO has been observed in the XRD pattern. It is also observed that the broadening of diffraction peaks is slightly decreased, and there is a minor increase in peak intensity was observed in fig. 1. Thus, it can understand that the substitution of Pb in the CdS lattice results in a slight increase in the volume of the unit cell, thereby producing a change (enhancement in a and c directions) in the lattice parameters as inferred by the shift in X-ray diffraction peak to the high angle side.



**Fig. 1: X-ray diffractograms of pure CdS, 4 mole%  $\text{Pb}^{2+}$  and 8 mole%  $\text{Pb}^{2+}$  doped CdS nanoparticles.**

The lattice parameters (a=b, c) of the CdS nanoparticles are calculated from the following equation,

$$\frac{1}{d^2} = \frac{4}{3} \left( \frac{h^2 + hk + k^2}{a^2} \right) + \frac{l^2}{c^2}$$

Where d is the inter-planar distance and h, k, l are the Miller indices of the respective plane. The observed structural data from the XRD measurements are summarized in table 1.

**Table 1. The observed structural data from XRD measurements**

Sample Name	Calculated parameters			Crystallite size nm
	a=b (Å)	c (Å)	Volume (Å <sup>3</sup> )	
CdS (JCPDS File No. 41-1049)	4.207	6.843	104.89	-
Pure CdS	4.193	6.816	103.78	14.3
4 mole% Pb doped CdS	4.214	6.836	105.13	15.7
8 mole% Pb doped CdS	4.226	6.847	105.89	18.3

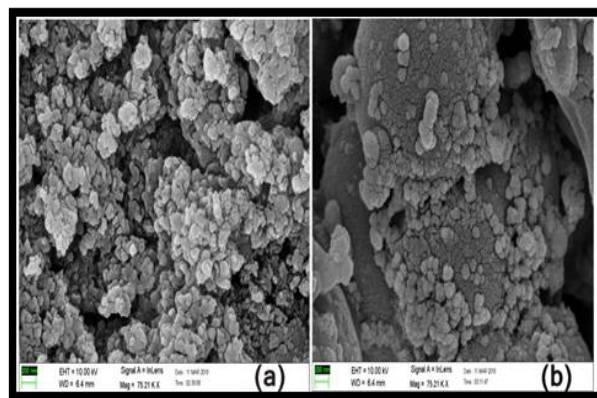
The crystallite sizes (D) of the pure CdS, 4 mole%  $\text{Pb}^{2+}$  and 8 mole%  $\text{Pb}^{2+}$  doped CdS nanoparticles are estimated from XRD pattern by using the Scherrer equation (Abd El-Sadek *et al.* 2019),

$$D = \frac{0.9 \lambda}{\beta \cos \theta}$$

Where D is the mean size (diameter) of the crystallite,  $\beta$  is the full width at half maximum of intensity (in radians),  $\lambda$  is the wavelength of the X-ray radiation used (1.540598 Å), and  $\theta$  is half the angle at which maximum intensity was observed. The estimated crystallite size of prepared nanoparticles is given in table 1, and it indicates the nanocrystalline nature of the samples. Also, the crystallite sizes of 4 and 8 mole%  $\text{Pb}^{2+}$  doped CdS nanoparticles are higher than that of pure CdS and is found to be increased with the increase in dopant concentration. The large atomic radius of Pb is one of the factors that contribute to the increase in crystallite size of these nanoparticles. This reveals the incorporation of  $\text{Pb}^{2+}$  into the host CdS matrix.

### 3.2 FESEM Analysis

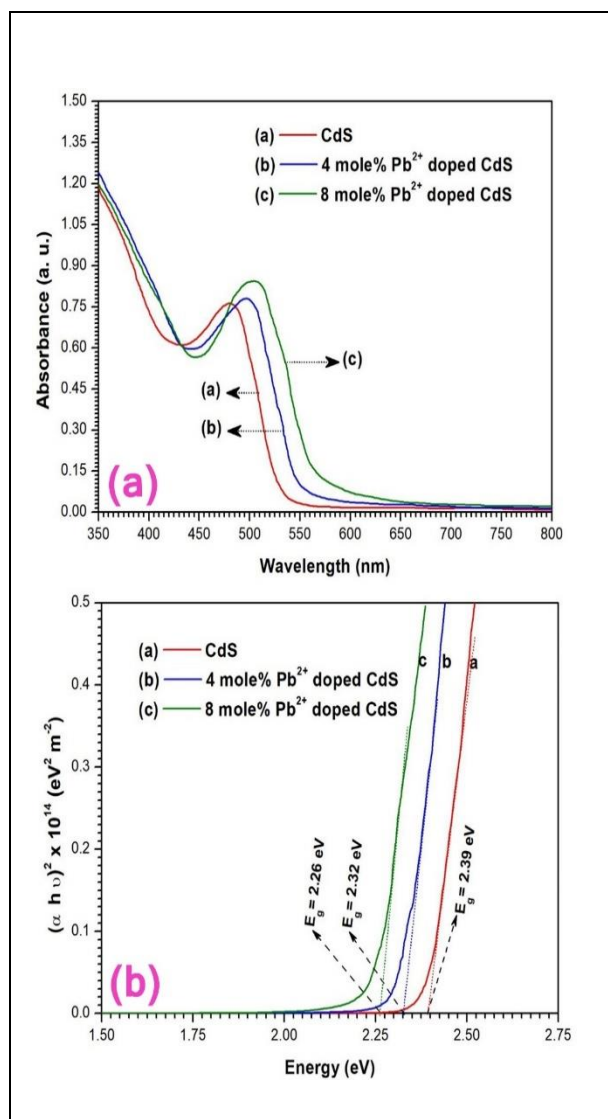
The field emission scanning electron microscopic structures of pure CdS and 8 mole%  $\text{Pb}^{2+}$  doped CdS nanoparticles is shown in fig. 2. From the FESEM microstructures, it can be seen that the pure CdS nanoparticles are composed of nanoparticles with a diameter of 25 nm to 35 nm, which is consistent with the values calculated by Scherrer's formula. It can also be seen that the  $\text{Pb}^{2+}$  doping in CdS matrices does not create much change following the morphology of the nanocrystals. However, particle size increases with the addition of dopant in the CdS lattices. The estimated particle size for 8 mole % Pb doped CdS nanoparticles is 40 -50 nm. Thus, it appears to be very big in size as compared to pure CdS nanoparticles.



**Fig. 2: FESEM microstructures of solvothermal synthesized (a) pure CdS nanoparticles, (b) 8 mole%  $\text{Pb}^{2+}$  doped CdS nanoparticles**

### 3.3 UV-Vis. Analysis

The variation of absorbance as a function of incident photon wavelength ( $\lambda$ ) for nanocrystalline pure CdS and different concentration of  $\text{Pb}^{2+}$  (4 mole% and 8 mole%) doped CdS nanoparticles are shown in fig. 3a. The corresponding incident photon energy ( $h\nu$ ) vs  $(\alpha h\nu)^2$  plot is given in fig. 3b. The absorption spectra of pure and doped CdS nanoparticles consist of short wavelength tail bands. This reveals that the particle size distribution is narrow. The increase in the intensity of absorption is due to increasing concentration of  $\text{Pb}^{2+}$  dopant from 4 mole% to 8 mole% in the host CdS lattices.



**Fig. 3: (a) UV-Vis. Absorbance spectra and (b) Optical bandgap measurement plots of pure and doped CdS nanoparticles**

The fundamental absorption edge in most semiconductor nanoparticles follows an exponential law.

The absorption coefficient has been reported to obey the following equation (Saravanan *et al.* 2012):

$$(\alpha h\nu)^{1/n} = A(h\nu - E_g)$$

where  $\nu$  is the frequency of the incident beam),  $A$  is a constant,  $E_g$  is optical band gap, and  $n$  is an exponent, which can be assumed to have values of  $1/2$ ,  $3/2$ ,  $2$  and  $3$  depending on the nature of electronic transition responsible for the absorption:  $n=1/2$  for allowed direct transition,  $n=3/2$  for forbidden direct transition,  $n=2$  for allowed indirect transition and  $n=3$  for forbidden indirect transition. The present system of pure and doped CdS semiconductor nanoparticles obeys the role of direct transition. The relation between the optical gap, optical absorption coefficient ( $\alpha$ ) and the energy ( $h\nu$ ) of the incident photon is given by (Saravanan *et al.* 2012),

$$(\alpha h\nu)^2 = A(h\nu - E_g)$$

The calculated optical band gap ( $E_g$ ) for pure CdS, 4 mole% Pb doped CdS and 8 mole% Pb doped CdS nanoparticles is calculated to be 2.39 eV, 2.32 eV and 2.26 eV, respectively. It is evident from the energy bandgap determination spectra that the value of  $E_g$  decreases with the addition of  $\text{Pb}^{2+}$  in the host CdS lattices and decreases with the increase of dopant concentration from 4 mole% to 8 mole%. This may be ascribed to the growth in grain size and growth in density of defect states.

### 4. CONCLUSIONS

Pure and doped CdS nanoparticles were synthesized successfully using a facile solvothermal process. The diffraction peaks obtained from X-ray diffraction patterns (XRD) of pure CdS, 4 mole%  $\text{Pb}^{2+}$  and 8 mole%  $\text{Pb}^{2+}$  doped CdS nanoparticles are found to have preferential orientations along (1 0 0), (0 0 2), (1 0 1), (1 0 2), (1 1 0), (1 0 3), (1 1 2), (0 0 4) and (2 0 3) plane, from which the observed peaks can be indexed to hexagonal phase of CdS with space group  $P6_3mc$ . Further, from the recorded spectra, one can understand that the degree of crystallinity improves with the increasing concentration of dopant in the host CdS lattices. More interestingly, when  $\text{Pb}^{2+}$  is added into the pure CdS, no diffraction peaks correspond to  $\text{PbS/CdO}$  have been observed in the XRD pattern. However, the broadening of the peak is slightly decreased, whereas the peak intensity is increased. Thus the lattice parameters and volume of the unit cell are slightly increased. This reveals that the  $\text{Pb}^{2+}$  ions are well incorporated within the host matrix. The estimated crystallite size of the  $\text{Pb}^{2+}$  doped CdS nanoparticles are higher than that of pure CdS



nanoparticles, and this is attributed to the higher ionic radii of Pb than that of parent CdS.

From the FESEM microstructures, it can be seen that the pure CdS nanoparticles are composed of nanoparticles with a diameter 25 nm to 35 nm and the particle size increases to 40–50 nm with the addition of dopant (8 mole % Pb) in the CdS lattices. The calculated optical band gap ( $E_g$ ) for pure CdS, 4 mole% Pb doped CdS and 8 mole% Pb doped CdS nanoparticles are found to be 2.39 eV, 2.32 eV and 2.26 eV, respectively.

The significant difference between the bandgap values of pure and doped nanoparticles is evidence for the incorporation of dopant in the parent CdS lattices. Further, the bandgap of these nanoparticles can be tuned well with a selection of suitable methods for synthesis. The solvothermal method leads to the agglomeration of particles due to the direct heating procedure. Undoubtedly the prepared pure and doped CdS nanoparticles synthesized by the simple solvothermal method are seen to yield good results without any additional impurities. A disadvantage of the present solvothermal method that we have observed is that the crystallite size over 10 nm obtained in the samples limits their applications due to the lack of the size quantization effect. The authors observed that a suitable futuristic method with pronounced control over crystallite size would open up new vistas for these synthesized materials.

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